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Tobacco and tobacco products — Determination of ammonia — Method using ion chromatographic analysis

Tabac et produits du tabac — Dosage de l'ammoniac — Méthode par chromatographie ionique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In 2013, the CORESTA (Cooperation Centre for Scientific Research Relative to Tobacco) Smokeless Tobacco Sub-Group (STS) conducted a collaborative study for the determination of ammonia (as ammonium ion) in tobacco and smokeless tobacco products (STP). Eleven laboratories participated in the study. The method provides baseline resolution for ammonium and sodium, which is an important consideration for matrices with a high sodium content, such as moist smokeless tobacco. The method was shown to be appropriate for the determination of ammonia in tobacco, smokeless tobacco products, and cigarette filler. The repeatability and reproducibility values of this method were assessed in accordance with ISO 5725-2:1994 and ISO/TR 22971:2005.

Tobacco and tobacco products — Determination of ammonia — Method using ion chromatographic analysis

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1 Scope

This document specifies a method for the determination of ammonia in tobacco and tobacco products using ion chromatographic analysis.

It is applicable to tobacco, processed tobaccos, smokeless tobacco products and cigarette filler. The ammonia calibration range specified in the method is from 0,100 µg/ml to 10,0 µg/ml. This range correlates to 10 µg/g to 1 000 µg/g ammonia in tobacco. Samples with higher levels of ammonia can be diluted prior to analysis to bring the samples within the calibration range.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
suppressor
device that reduces the background conductance of the eluent

Note 1 to entry: This can improve the signal-to-noise ratio.

3.2
portioned product
tobacco product that is pre-portioned into a serving size

EXAMPLE A snus pouch.

4 Principle

The ammonia content of tobacco or tobacco products, including smokeless tobacco products (STP) and cigarette filler, is determined by extraction into a sulfuric acid solution. Ion chromatographic analysis is used to separate ammonium ion from other cationic species. The response of ammonium ion is measured using a conductivity detector and quantified against an external standard calibration. Results are reported as ammonia.

5 Apparatus

Usual laboratory apparatus and equipment for use in preparation of samples and standards, and in particular the following items. All labware shall be cleaned before use to avoid any contamination.

5.1 Analytical balance, capable of measuring to at least four decimal places (gram).

5.2 Syringe filter, 0,45 µm nylon, or equivalent.

5.3 Polypropylene volumetric flasks, of capacities 100 ml, 250 ml and 1 000 ml.

5.4 Mechanical pipettes with disposable plastic tips, 10 µl to 1 000 µl.

5.5 Laboratory shaker.

5.6 Polypropylene sample extraction vessels with caps, 100 ml approximate volume.

5.7 Weak cation exchange column of mid-capacity, (250 mm × 4 mm, non-metallic), approximately 2,8 milliequivalents per column.

EXAMPLE Thermo Scientific IonPac® CS12A[1)], or equivalent.

Other column(s) might be suitable for use with this method; however, laboratories shall verify that sodium is sufficiently resolved from ammonium, in the test samples, before use.

5.8 Ion chromatograph (IC), consisting of a conductivity detector, suppressor and data collection system.

NOTE This method can be used without a suppressor, however, this will result in a reduced signal-to-noise ratio unless the eluent and chromatographic conditions are modified. As a consequence, it might not be possible to analyse samples with low levels of ammonia such as certain Virginia or Oriental tobaccos.

5.9 Eluent degassing unit, recommended.

6 Reagents

Use only reagents of recognized analytical grade.

6.1 Ammonium sulphate, $[(NH_4)_2SO_4]$, >99 % purity (25,78 % ammonia); alternatively a certified solution of 1 000 µg/ml ammonia may be used.

6.2 Sulfuric acid, (H_2SO_4), >96 % purity.

6.3 Methanesulfonic acid (MSA), >99 % purity.

1) Thermo Scientific IonPac® CS12A cation exchange analytical column is the trade name of a suitable product available commercially. This information is given for the convenience of the users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

7 Preparation

7.1 Preparation of labware

Labware shall be cleaned and dried in a manner which ensures that contamination does not occur.

It is important that all possible sources of contamination which might interfere with the analytical process are removed from the work area.

7.2 Preparation of solutions

7.2.1 Sulfuric acid, 0,012 5 mol/l (0,025 N), used for standards and extraction solution.

Carefully add 1,3 g of sulfuric acid to approximately 600 ml of deionised water in a 1 l polypropylene volumetric flask.

Mix and dilute to 1 l with deionised water.

7.2.2 Eluent A: 10 mM methanesulfonic acid (MSA).

Carefully add 650 µl of methanesulfonic acid (MSA) to approximately 600 ml of deionised water in a 1 l polypropylene volumetric flask.

Mix and dilute to 1 l with deionised water.

7.2.3 Eluent B: 40 mM methanesulfonic acid (MSA).

Carefully add 2,60 ml of methanesulfonic acid (MSA) to approximately 600 ml of deionised water in a 1 l polypropylene volumetric flask.

Mix and dilute to 1 l with deionised water.

7.3 Preparation of standards

7.3.1 Prepare a series of seven working calibration standard solutions as described in Table 1.

A minimum of seven standards are required because the calibration curve is quadratic.

NOTE 1 Quantitation is obtained from an external standard calibration using the peak area response of ammonium (NH_4^+) as ammonia (NH_3).

NOTE 2 All calculations are based on the ammonia molar mass.

NOTE 3 The stock and working standard solutions are stable for approximately 30 days when stored at 4 °C.

7.3.2 Ammonia stock solution, 100 µg/ml.

Weigh approximately 0,097 g of ammonium sulphate into a 250 ml polypropylene volumetric flask.

Record the exact weight in order to accurately calculate the actual concentration.

Add 0,012 5 mol/l (0,025 N) sulfuric acid and mix thoroughly.